How to Boost Silicone Rubber Compounds

Author: Nicole Holzmayr
Status Quo

Addition of inactive fillers to silicone rubber

- cost reduction
- optimization of processing properties
- improvement of mechanical properties
Filler Selection

Silicone rubber

Silfit Z 91
Focus: color, cost/performance ratio

Aktisil Q
Focus: mechanical properties
Topic Selection

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Silfit Z 91

- basic properties
- flame retardant compounds
Boosting silicone rubber with Silfit Z 91
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Quartz flour

Silfit Z 91
# Formulation

Typical for extrusion

<table>
<thead>
<tr>
<th>Ingredient</th>
<th>in phr</th>
</tr>
</thead>
<tbody>
<tr>
<td>Filler as indicated</td>
<td>as indicated</td>
</tr>
<tr>
<td>Curing Agent E</td>
<td>1.5</td>
</tr>
<tr>
<td>Bis-2,4-(dichlorobenzoyl)-peroxide</td>
<td></td>
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<tr>
<td>Elastosil R 401/40</td>
<td>100</td>
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</table>

<table>
<thead>
<tr>
<th>Condition</th>
<th>Details</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cure conditions</td>
<td>5 min. / 115 °C</td>
</tr>
<tr>
<td>Post-cure</td>
<td>4 h / 200 °C</td>
</tr>
<tr>
<td>Immersion in reference oil IRM 903</td>
<td>72 h / 150 °C</td>
</tr>
</tbody>
</table>
Blooming

after approx. 7 months, post-cured; specimens black-colored

<table>
<thead>
<tr>
<th></th>
<th>Base cpd.</th>
<th>25 phr</th>
<th>50 phr</th>
<th>75 phr</th>
</tr>
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<tbody>
<tr>
<td>Silfit Z 91</td>
<td></td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>Quartz flour</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
COLOR

ISO 7724

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Silfit Z 91

Basic properties

**SUMMARY**

![Graphs showing color changes with different phr levels for Silfit Z 91 and Quartz flour.](image)
IRM 903, 72 h / 150°C
Change of Tensile Strength

Post-cure, 4 h / 200°C

% change

Filler in phr

Silfit Z 91
Quartz flour

Base cpd.

25
50
75

IRM 903, 72 h / 150°C
Change of Tensile Strength

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IRM 903, 72 h / 150°C
Change of Elongation at Break

Post-cure, 4 h / 200°C

Silfit Z 91
Quartz flour

%
Silfit Z 91 in Silicone Rubber

Good extrusion and processing behavior
- lower abrasivity
- less formation of dust
- equal output
- prevention of blooming

Comparable mechanical properties along with
- higher color neutrality and brightness
- improved oil resistance

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Boosting ATH-filled, flame-retardant silicone rubber with Silfit Z 91
Status Quo

Active flame retardancy
aluminium hydroxide - ATH

- thermal decomposition of ATH into Al₂O₃ and H₂O

Passive flame retardancy
Neuburg Siliceous Earth

- mainly dilution of flammable gases by reduction of the polymer portion with increasing filler loading
What did we do?

High loading of ATH necessary to promote flame retardancy

Decreasing mechanical properties

Approach

ATH silanized

ATH without silane + Silfit Z 91
Influence on Flame Retardancy

ATH + Silfit Z 91 = ?

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## Formulation

<table>
<thead>
<tr>
<th>in phr</th>
<th>ATH</th>
<th>ATH silanized</th>
<th>ATH Silfit Z 91</th>
</tr>
</thead>
<tbody>
<tr>
<td>ATH</td>
<td>100</td>
<td>-</td>
<td>70</td>
</tr>
<tr>
<td>ATH silanized</td>
<td>-</td>
<td>100</td>
<td>-</td>
</tr>
<tr>
<td>Silfit Z 91</td>
<td>-</td>
<td>-</td>
<td>30</td>
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<tr>
<td>Curing Agent E</td>
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<tr>
<td>Bis-2,4-(dichloro-benzoyl)-peroxide</td>
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<tr>
<td>Elastosil R 401/40</td>
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<td>100</td>
<td></td>
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</table>

**Cure conditions**: 5 min. / 115 °C

**Post-cure**: 4 h / 200 °C
LOI
Limiting Oxygen Index

ISO 4589, no post-cure

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Silfit Z 91
Flame retardant comp.

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Heat Release Rate

ISO 5660, 50 kW/m²

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100 phr
ATH

100 phr
ATH silanized

70 + 30 phr
Silfit Z 91

ATH

Smoke Density

ISO 5660, 50 kW/m²

- ATH 100 phr
- ATH silanized 100 phr
- Silfit Z 91 70 + 30 phr
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Flame retardant comp.

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**Tensile Strength**

*DIN 53 504, S2*

![Graph showing tensile strength for different materials and curing methods.](image)

- **ATH**: 4.5 MPa (100 phr)
- **ATH silanized**: 7.0 MPa (100 phr)
- **ATH Silfit Z 91**: 6.0 MPa (70 + 30 phr)

- **Press-cured**
- **Post-cured**
Elongation at Break

DIN 53 504, S2

- ATH
  - 100 phr
- ATH silanized
  - 100 phr
- ATH Silfit Z 91
  - 70 + 30 phr

EXPERIMENTAL

RESULTS
- Silfit Z 91
- Flame retardant comp.

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Teard Resistance

DIN ISO 34-1, Graves

<table>
<thead>
<tr>
<th></th>
<th>ATH 100 phr</th>
<th>ATH silanized 100 phr</th>
<th>ATH Silfit Z 91 70 + 30 phr</th>
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<tbody>
<tr>
<td>press-cured</td>
<td>8</td>
<td>6</td>
<td>12</td>
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<tr>
<td>post-cured</td>
<td>7</td>
<td>4</td>
<td>10</td>
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</table>

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Silfit Z 91

Flame retardant comp.

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Compression Set
24 h / 175 °C

DIN ISO 815-1 B, cooling method A, 25 % defl.

![Graph showing compression set results with different materials and curing methods.](image-url)
Combination of ATH with Silfit Z 91

Unaffected outstanding flame retardancy

- LOI
- heat release rate
- smoke density

Good mechanical properties

- good tensile strength
- good elongation at break
- high tear resistance
- reduced compression set
Topic Selection

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Aktisil Q

- basic properties
- flame retardant compounds
- combination with untreated quartz flour
Boosting silicone rubber with Aktisil Q
Quartz flour
vinyl-silane treated

Aktisil Q
methacryl-silane treated
**Formulation**

*typical for extrusion*

<table>
<thead>
<tr>
<th>Component</th>
<th>in phr</th>
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<tr>
<td>Filler</td>
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<tr>
<td>Curing Agent E</td>
<td>1.5</td>
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<tr>
<td>Bis-2,4-(dichlorobenzoyl)-peroxide</td>
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<tr>
<td>Immersion in reference oil IRM 903</td>
<td>72 h / 150 °C</td>
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<tr>
<td>Extrusion</td>
<td>Hose, 50 rpm 25 °C</td>
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</table>
Collapse resistance

Quartz flour VST  Aktisil Q

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Aktisil Q
Basic properties

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Blooming

after 4 weeks, specimens black-colored

Base cpd.  Quartz flour VST  Aktisil Q

post-cured, 4 h / 200 °C  no post-cure

even after 1 year no blooming detectable with Aktisil Q
Compression Set
24 h / 175 °C

DIN ISO 815-1 B, cooling method A, 25 % defl.

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IRM 903, 72 h / 150°C
Volume change

Post-cured

<table>
<thead>
<tr>
<th>Base cpd.</th>
<th>20</th>
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<th>40</th>
<th>50</th>
<th>60</th>
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<tbody>
<tr>
<td>Filler in phr</td>
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<td>75</td>
<td>50</td>
<td>25</td>
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</table>

Aktisil Q
Quartz flour VST

SUMMARY
IRM 903, 72 h / 150°C
Change of Tensile Strength

Post-cured

<table>
<thead>
<tr>
<th>%</th>
<th>Base cpd.</th>
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<th>50</th>
<th>75</th>
<th>100</th>
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Aktisil Q
Quartz flour VST

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IRM 903, 72 h / 150°C
Tensile Strength

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Aktisil Q
Quartz flour VST

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Aktisil Q in Silicone Rubber

**outstanding extrusion and processing behavior**
- lower abrasivity than quartz flour and diatomaceous earth
- improved collapse resistance
- increased output
- reduced stickiness
- prevention of blooming

**improved mechanical properties**
- higher hardness
- reduced compression set, esp. press-cured
- comparable hot air resistance
- increased oil resistance
Boosting ATH-filled, flame retardant silicone rubber with Aktisil Q
Status Quo

Active flame retardancy
aluminium hydroxide - ATH

- thermal decomposition of ATH into $\text{Al}_2\text{O}_3$ and $\text{H}_2\text{O}$

Passive flame retardancy
Neuburg Siliceous Earth

- mainly dilution of flammable gases by reduction of the polymer portion with increasing filler loading
What did we do?

High loading of ATH necessary to promote flame retardancy

Decreasing mechanical properties

Approach

ATH silanized

ATH without silane + Aktisil Q
Influence on Flame Retardancy

ATH + Aktisil Q = ?
## Formulation

<table>
<thead>
<tr>
<th></th>
<th>ATH</th>
<th>ATH silanized</th>
<th>ATH Aktisil Q</th>
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<td><strong>in phr</strong></td>
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<td>Elastosil R 401/40</td>
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<td><strong>Cure conditions</strong></td>
<td>5 min. / 115 °C</td>
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<tr>
<td><strong>Post-cure</strong></td>
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<td></td>
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<tr>
<td></td>
<td>4 h / 200 °C</td>
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LOI
Limiting Oxygen Index

ISO 4589, no post-cure

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<tr>
<td>ATH silanized</td>
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<tr>
<td>ATH Aktisil Q</td>
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Aktisil Q
Flame retardant comp.

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Aktisil Q
Flame retardant comp.

SUMMARY

Heat Release Rate

ISO 5660, 50 kW/m²

ATH 100 phr
ATH silanized 100 phr
ATH Aktisil Q 70 + 30 phr
Smoke Density

ISO 5660, 50 kW/m²

- ATH
  - 100 phr
- ATH silanized
  - 100 phr
- Aktisil Q
  - 70 + 30 phr
Tensile Strength

DIN 53 504, S2

- ATH 100 phr
- ATH silanized 100 phr
- Aktisil Q 70 + 30 phr

- Press-cured
- Post-cured

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Elongation at Break

DIN 53 504, S2

- ATH
  - 100 phr
- ATH silanized
  - 100 phr
- ATH Aktisil Q
  - 70 + 30 phr

press-cured  post-cured
**Tear Resistance**

DIN ISO 34-1, Graves

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**RESULTS**

Aktisil Q
Flame retardant comp.

**SUMMARY**
Compression Set
24 h / 175 °C

DIN ISO 815-1 B, cooling method A, 25 % defl.

- **ATH**
  - 100 phr

- **ATH silanized**
  - 100 phr

- **ATH Aktisil Q**
  - 70 + 30 phr

**Comparison:**
- Press-cured
- Post-cured

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- Aktisil Q
- Flame retardant comp.

**SUMMARY**
Combination of ATH with Aktisil Q

Unaffected outstanding flame retardancy
- LOI
- heat release rate
- smoke density

Improved mechanical properties
- high tensile strength
- good elongation at break
- high tear resistance
- markedly reduced compression set
Silicone rubber filled with non-surface-treated quartz flour boosted with Aktisil Q
Status Quo

**Compounds designed for moldings**
- peroxides: Curing Agent C6 or Perkadox BC-40S-ps
- polymer: 40 Shore A base compound

**Fillers**
- quartz flour, untreated
- typical loading: 25 phr

**Problem**
- insufficient physical properties for some applications

**Approach**
- addition of small amount of Aktisil Q
- combination of 30 and 40 Shore A base compound to arrive at a comparable hardness
Status Quo

Sikron SF 600
Quarzwerke, GER

- typically used in the European area

MIN-U-SIL 5
U.S. Silica, USA

- typically used in the American area
### Formulation – Sikron SF 600

Typical for moldings

<table>
<thead>
<tr>
<th></th>
<th>Curing Agent C6</th>
<th>Perkadox BC-40S-ps</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Sikron</td>
<td>Sikron</td>
</tr>
<tr>
<td></td>
<td>Sikron</td>
<td>Aktisil Q</td>
</tr>
<tr>
<td></td>
<td>Sikron</td>
<td>Sikron</td>
</tr>
<tr>
<td></td>
<td>Sikron</td>
<td>Aktisil Q</td>
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<table>
<thead>
<tr>
<th></th>
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<th>S**</th>
<th>S**</th>
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<tbody>
<tr>
<td></td>
<td>25</td>
<td>12.5</td>
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<td>Curing Agent C6*</td>
<td>1.2</td>
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<td>Perkadox BC-40S-ps**</td>
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<table>
<thead>
<tr>
<th>Cure conditions</th>
<th>5 min. / 165 °C</th>
<th>5 min. / 180 °C</th>
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</thead>
<tbody>
<tr>
<td>Post-cure</td>
<td></td>
<td>4 h / 200 °C</td>
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<tr>
<td>Immersion in</td>
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<td>72 h / 150 °C</td>
</tr>
<tr>
<td>reference oil IRM 903</td>
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<td></td>
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</tbody>
</table>

* Curing Agent C6: 2,5-Bis-(t-butylperoxy)-2,5-dimethylhexane
** Perkadox BC-40S-ps: Dicumyl peroxide
Tear Resistance

DIN ISO 34-1 Bb, Graves; post-cured

Curing Agent C6

<table>
<thead>
<tr>
<th>Material</th>
<th>N/mm</th>
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<tbody>
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<td>Sikron SF 600</td>
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<td>Aktisil Q</td>
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Perkadox BC-40S-ps

<table>
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<th>Material</th>
<th>N/mm</th>
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<td>Aktisil Q</td>
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Boosting quartz flour

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Compression Set
24 h / 175 °C

DIN ISO 815-1 B, cooling method A, 25 % defl.; post-cured
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Boosting quartz flour

SUMMARY
## Formulation – MIN-U-SIL 5

**typical for moldings**

<table>
<thead>
<tr>
<th></th>
<th>Curing Agent C6</th>
<th>Perkadox BC-40S-ps</th>
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<tbody>
<tr>
<td><strong>MIN-U-SIL</strong></td>
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<tr>
<td>Aktisil Q</td>
<td><strong>25</strong></td>
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<tr>
<td>MIN-U-SIL 5</td>
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* Curing Agent C6: 2,5-Bis-(t-butylyperoxy)-2,5-dimethylhexane
** Perkadox BC-40S-ps: Dicumyl peroxide
Tear Resistance

DIN ISO 34-1 Bb, Graves; post-cured

Curing Agent C6 | Perkadox BC-40S-ps

<table>
<thead>
<tr>
<th>MIN-U-SIL 5</th>
<th>MIN-U-SIL 5 Aktisil Q</th>
<th>MIN-U-SIL 5</th>
<th>MIN-U-SIL 5 Aktisil Q</th>
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N/mm

INTRODUCTION
EXPERIMENTAL
RESULTS
Aktisil Q
Boosting quartz flour
SUMMARY
Compression Set
24 h / 175 °C

DIN ISO 815-1 B, cooling method A, 25 % defl.; post-cured

Curing Agent C6

Perkadox BC-40S-ps

%  

MIN-U-SIL 5  MIN-U-SIL 5 Aktisil Q  MIN-U-SIL 5  MIN-U-SIL 5 Aktisil Q

Aktisil Q

Boosting quartz flour
Combination of Quartz Flour with Aktisil Q

Sikron SF 600
- comparable tear resistance
- improved compression set
- improved oil resistance
- potential reduction of compound costs of -5 %

MIN-U-SIL 5
- improved tear resistance
- improved compression set
- potential reduction of compound costs of -5 %
Conclusion

Aktisil Q
Fillers for a wide range of applications in silicone rubber

Silfit Z 91
Previous tests showed:

- 25 phr Aktisil Q
- compounds designed for molded parts
- exceed tear resistance of base compound

Further investigations will follow
Thank you very much for your attention!

For more information please visit our website:
www.hoffmann-mineral.com